Supporting Information for:

Toxicity of Hydraulic Fracturing Wastewater from Black Shale Natural-Gas Wells Influenced by Well Maturity and Chemical Additives

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This Supporting Information contains the following:

Major ion concentrations (mg/L) of salt matched control (Table S1), Bioluminescence inhibition assay data (Table S2), NAC-thiol reactivity assay data (Table S3), Hydraulic fracturing fluid composition details for M-4 and M-5 wells in Marcellus Shale and U-6 and U-7 wells in the Utica-Point Pleasant Formation (Table S4-S7), organic chemistry data (Table S8) and iodinated organic ions data (Table S9); Experimental factors (Figure S1) and Organic chemistry trends (Figure S2)

I. Supporting Tables

Table S1. Major ion concentrations (mg/L) of salt matched control that mimicked the chemistry of Appalachian shale FPW.

Constituents	Measured Conc. (mg/L)
Ca^{+2}	8000
Mg^{+2}	748.75
Na^{+}	20185
K^{+}	660
Cl^{-}	48113

Table S2. Bioluminescence Inhibition Assay Data

Type of sample	Well #	Approximate Time After Flowback Began (Days)	Sampling Date	Days after Fracturing	TDS (ppt)	DOC (mg/L)	Dilution Factor	pH	BLIA Inhibitory Effect (S)	BLIA Inhibitory Effect (SF)	TU_{50}
MARCELLUS SHALE											
M-4 fracture fluid (Kill fluid)	M-4	NA	3/3/2017	NA	19.1	NA	1	9.22	89.14	88.22	51.28
M-4 Drill mud	M-4	NA	8/28/2015	NA	124.9	NA	70	8.99	52.05	52.49	2.10
M-4 Sidewall mud	M-4	NA	9/3/2015	NA	104.0	NA	6	8.17	37.36	44.86	1.41
M-4 FPW	M-4	2	12/11/2015	25	17.50	312.2	4	6.4	5.25	0.37	0.030
M-4 FPW	M-4	13	12/22/2015	36	25.20	84.9	3	6.36	6.96	5.34	0.656
M-4 FPW	M-4	56	2/3/2016	79	55.30	98.5	5	5.99	8.06	0.8	0.006
M-4 FPW	M-4	70	2/17/2016	93	65.10	68.64	5	5.86	11.61	16.18	0.396
M-4 FPW	M-4	182	6/8/2016	205	143.50	59.67	5	6.35	6.73	4.43	0.002
M-4 FPW	M-4	280	9/14/2016	303	212.10	60.96	5	6.05	11.13	5.35	0.006
M-4 FPW	M-4	406	1/18/2017	429	300.30	416.15	6	5.42	-11.15	-22.36	-
M-4 FPW	M-4	490	4/12/2017	513	359.10	104.1	6	5.22	-15.78	-25.47	-
M-4 FPW	M-4	641	8/16/2017	664	464.80	NA	6	5.28	-17.21	-30.48	-
M-4 FPW	M-4	764	12/13/2017	787	550.90	NA	6	5.39	-24.98	-26.54	-
M-5 FPW	M-5	2	12/11/2015	36	25.20	68.91	3	6.3	33.61	30.76	3.68
M-5 FPW	M-5	9	12/18/2015	43	30.10	50.86	3	6.42	37.31	26.96	1.78
M-5 FPW	M-5	56	2/3/2016	90	63.00	159.9	4	6.21	29.11	20.24	0.43
M-5 FPW	M-5	70	2/17/2016	104	72.80	43.35	5	6.1	21.98	28.5	0.15
M-5 FPW	M-5	119	4/6/2016	153	107.10	41.96	5	6.27	26.69	10.24	1.16
M-5 FPW	M-5	182	6/8/2016	216	151.20	49.92	6	6.38	5.63	6.18	0.00
M-5 FPW	M-5	280	9/14/2016	314	219.80	50.2	6	6.25	-1.2	-30.88	0.00
M-5 FPW	M-5	764	12/13/2017	798	558.60	NA	4	5.72	6.08	-7.99	-
UTICA SHALE											
SW Fresh Water Tank	-	N/A	5/7/2015	N/A	0.14903	NA	-	8.541	-12.06	-28.62	-
SW Fresh Water Tank	-	N/A	5/14/2015	N/A	0.16387	NA	-	7.68	-9.88	-27.66	-
SW Produced Water Additive	-	N/A	5/14/2015	N/A	127.82	NA	7	6.323	4.52	-8.68	-

SW Produced Water Additive	-	N/A	5/29/2015	N/A	129.08	NA	7	5.71	1.54	13.18	-
U-6 FPW	U-6	1	7/14/2015	38	89.88	83.3	5	6.588	4.36	27.8	0.103
U-6 FPW	U-6	9	7/23/2015	46	109.27	64.06	6	6.33	9.72	16.78	0.067
U-6 FPW	U-6	16	7/30/2015	54	113.54	60.74	6	6.36	5.42	13.12	0.192
U-6 FPW	U-6	22	8/5/2015	60	117.6	83.82	6	6.32	21.87	12.31	0.531
U-6 FPW	U-6	30	8/13/2015	68	119.42	59.78	7	6.28	-24.87	-28.63	-
U-6 FPW	U-6	58	9/10/2015	96	130.55	48.62	7	6.22	-26.29	-25.16	-
U-6 FPW	U-6	87	10/8/2015	124	86.8	36.62	5	3.97	-26.17	-18.53	-
U-6 FPW	U-6	122	11/12/2015	159	111.3	50.29	7	4.5	-32.83	-32.14	-
U-6 FPW	U-6	392	8/8/2016	460	322	22.904	7	5.31	-18.05	-51.79	-
U-7 FPW	U-7	1	7/14/2015	38	128.73	153.9	7	5.97	-11.32	-39.14	-
U-7 FPW	U-7	9	7/23/2015	46	114.03	80.76	6	6.32	-19.73	-29.64	-
U-7 FPW	U-7	16	7/30/2015	54	120.05	61.41	7	6.2	-39.72	-46.68	-
U-7 FPW	U-7	30	8/13/2015	68	128.66	51.96	7	6.2	-47.79	-57.31	-
U-7 FPW	U-7	58	9/10/2015	96	135.87	55.23	7	6.15	-24.97	-17.72	-
U-7 FPW	U-7	87	10/8/2015	124	86.8	36.2	7	5.58	-26.58	-25.18	-
U-7 FPW	U-7	122	11/12/2015	159	111.3	40.46	7	5.03	-55.65	-62.91	-
U-7 FPW	U-7	392	8/8/2016	460	322	22.8	7	5.54	-59.28	-53.92	-

Table S3. NAC Thiol Reactivity Data

Type of sample	Well #	Approximate Time After Flowback Began (Days)	Sampling Date	Days after Fracturing	Concentration Factor	NAC-Thiol Assay: Response as the Mean Percent of the Negative Control (±SE)
MARCELLUS SHALE						
M-4 fracture fluid	M-4	NA	11/11/2015	NA	25	85.81
Input river	M-4	NA	11/11/2015	NA	200	89.75
M-4 FPW	M-4	4	12/13/2015	27	200	82.98
M-4 FPW	M-4	13	12/22/2015	36	200	80.5
M-4 FPW	M-4	56	2/3/2016	79	200	77.63
M-4 FPW	M-4	70	2/17/2016	93	200	78.27
M-4 FPW	M-4	182	6/8/2016	205	200	77.79
M-4 FPW	M-4	280	9/14/2016	303	200	89.67
M-4 FPW	M-4	406	1/18/2017	429	200	80.04
M-4 FPW	M-4	490	4/12/2017	513	200	91.28
M-4 FPW	M-4	641	8/16/2017	664	200	91.65
M-4 FPW	M-4	764	12/13/2017	787	200	96.08
M-5 fracture fluid	M-5	NA	11/5/2015	NA	25	90.45
Input river	M-5	NA	11/5/2015	NA	200	90.65
M-5 FPW	M-5	9	12/18/2015	43	200	78.97
M-5 FPW	M-5	36	1/14/2016	70	200	87.31
M-5 FPW	M-5	56	2/3/2016	90	200	85.5
M-5 FPW	M-5	70	2/17/2016	104	200	86.46
M-5 FPW	M-5	119	4/6/2016	153	200	92.3
M-5 FPW	M-5	182	6/8/2016	216	200	77.6
M-5 FPW	M-5	280	9/14/2016	314	200	99.09
M-5 FPW	M-5	764	12/13/2017	798	200	94.47

Table S4. Hydraulic fracturing fluid composition details for M-4 well in Marcellus Shale

Job Start Date:	11/6/2015
Job End Date:	11/15/2015
State:	West Virginia
County:	Monongalia
True Vertical Depth:	7,483
Total Base Water Volume (gal):	10,647,966
Total Base Non Water Volume:	0

Hydraulic Fracturing Fluid Composition:

Trade Name	Supplier	Purpose	Ingredients	Chemical Abstract Service Number (CAS #)	Maximum Ingredient Concentration in Additive (% by mass)**	Maximum Ingredient Concentration in HF Fluid (% by mass)**	Comments
Ingredients shown above are subject to 29 CFR 1910.1200(i) and appear on Material Safety Data Sheets (MSDS). Ingredients shown below are Non-MSDS.							
Proppant Transport	Schlumberger	Corrosion Inhibitor, Scale Inhibitor, Biocide, AntiFoam Agent, Acid, Breaker, Gelling Agent, Friction Reducer, Iron Control Agent, Fluid Loss Additive					
			Water (Including Mix Water Supplied by Client)*	NA		87.63568	
			Quartz, Crystalline silica	14808-60-7	99.06784	12.21724	
			Hydrochloric acid	7647-01-0	0.66726	0.08228	
			Ammonium sulfate	7783-20-2	0.06845	0.00844	
			Guar gum	9000-30-0	0.05865	0.00724	
			Acrylamide, 2-acrylamido-2-methylpropanesulfonic acid, sodium salt polymer	38193-60-1	0.05052	0.00623	
			Glutaraldehyde	111-30-8	0.02831	0.00349	
			Ethanol, 2,2',2''-nitrilotris-, 1,1',1''-tris(dihydrogen phosphate), sodium salt	68171-29-9	0.00971	0.00120	
			Diammonium peroxidisulphate	7727-54-0	0.00601	0.00074	

			Polymer of 2-acrylamido-2-methylpropanesulfonic acid sodium salt and methyl acrylate	136793-29-8	0.00541	0.00067	
			Alkyl(c12-16) dimethylbenzyl ammonium chloride	68424-85-1	0.00506	0.00062	
			Sodium erythorbate	6381-77-7	0.00436	0.00054	
			Trisodium ortho phosphate	7601-54-9	0.00427	0.00053	
			Urea	57-13-6	0.00332	0.00041	
			Polypropylene glycol	25322-69-4	0.00294	0.00036	
			Methanol	67-56-1	0.00252	0.00031	
			Fatty acids, tall-oil	61790-12-3	0.00156	0.00019	
			Thiourea, polymer with formaldehyde and 1-phenylethanone	68527-49-1	0.00129	0.00016	
			Ethylene Glycol	107-21-1	0.00121	0.00015	
			Non-crystalline silica (impurity)	7631-86-9	0.00084	0.00010	
			Vinylidene chloride/methylacrylate copolymer	25038-72-6	0.00080	0.00010	
			Sodium sulfate	7757-82-6	0.00078	0.00010	
			Alcohols, C14-15, ethoxylated (7EO)	68951-67-7	0.00061	0.00008	
			Ethanol	64-17-5	0.00061	0.00007	
			Propargyl alcohol	107-19-7	0.00041	0.00005	
			2-Propenamid (impurity)	79-06-1	0.00017	0.00002	
			Hexadec-1-ene	629-73-2	0.00014	0.00002	
			1-Octadecene (C18)	112-88-9	0.00007	0.00001	
			Dimethyl siloxanes and silicones	63148-62-9	0.00005	0.00001	
			Tetrasodium ethylenediaminetetraacetate	64-02-8	0.00009	0.00001	
			Dodecamethylcyclohexasiloxane	540-97-6			
			Siloxanes and silicones, dimethyl, reaction products with silica	67762-90-7	0.00001		
			Octamethylcyclotetrasiloxane	556-67-2			
			poly(tetrafluoroethylene)	9002-84-0	0.00001		
			Formaldehyde	50-00-0	0.00001		
			Copper(II) sulfate	7758-98-7			
			Decamethyl cyclopentasiloxane	541-02-6			
			Magnesium silicate hydrate (talc)	14807-96-6	0.00002		
FR Pro 150	ECM	Friction Reduction					
			Water	7732-18-5	50.00000	0.01575	
			Polyacrylamide-co-acrylic acid	9003-06-9	32.00000	0.01008	
			Sodium Chloride	7647-14-5	15.00000	0.00472	
			Alcohol Ethoxylate Surfactants	Trade	5.00000	0.00157	
			Petroleum Distillate	64742-47-8	25.00000		

* Total Water Volume sources may include fresh water, produced water, and/or recycled water
** Information is based on the maximum potential for concentration and thus the total may be over 100%

Note: For Field Development Products (products that begin with FDP), MSDS level only information has been provided.
Ingredient information for chemicals subject to 29 CFR 1910.1200(i) and Appendix D are obtained from suppliers Material Safety Data Sheets (MSDS)

Table S5. Hydraulic fracturing fluid composition details for M-5 well in Marcellus Shale

Job Start Date	10/28/2015
Job End Date	11/5/2015
State	West Virginia
County	Monongalia
True Vertical Depth	7,530
Total Base Water Volume (gal)	9,961,350
Total Base Non Water Volume	0

Hydraulic Fracturing Fluid Composition:

Trade Name	Supplier	Purpose	Ingredients	Chemical Abstract Service Number (CAS #)	Maximum Ingredient Concentration in Additive (% by mass)**	Maximum Ingredient Concentration in HF Fluid (% by mass)**	Comments
Ingredients shown above are subject to 29 CFR 1910.1200(i) and appear on Material Safety Data Sheets (MSDS). Ingredients shown below are Non-MSDS.							
Proppant Transport	Schlumberger	Corrosion Inhibitor, Scale Inhibitor, Biocide, Acid, Breaker, Gelling Agent, Friction Reducer, Iron Control Agent, Fluid Loss Additive , Propping Age					
			Water (Including Mix Water Supplied by Client)*	NA		87.58016	
			Quartz, Crystalline silica	14808-60-7	98.77034	12.26228	
			Hydrochloric acid	7647-01-0	0.90405	0.11223	
			Ammonium sulfate	7783-20-2	0.12127	0.01506	
			Acrylamide, 2-acrylamido-2-methylpropanesulfonic acid, sodium salt polymer	38193-60-1	0.08951	0.01111	
			Glutaraldehyde	111-30-8	0.03083	0.00383	
			Guar gum	9000-30-0	0.02213	0.00275	
			Polymer of 2-acrylamido-2-methylpropanesulfonic acid sodium salt and methyl acrylate	136793-29-8	0.00959	0.00119	

			Ethanol, 2,2',2''-nitrilotris-, 1,1',1''-tris(dihydrogen phosphate), sodium salt	68171-29-9	0.00943	0.00117	
			Sodium erythorbate	6381-77-7	0.00589	0.00073	
			Urea	57-13-6	0.00589	0.00073	
			Alkyl(c12-16) dimethylbenzyl ammonium chloride	68424-85-1	0.00551	0.00068	
			Trisodium ortho phosphate	7601-54-9	0.00415	0.00051	
			Methanol	67-56-1	0.00332	0.00041	
			Fatty acids, tall-oil	61790-12-3	0.00210	0.00026	
			Thiourea, polymer with formaldehyde and 1-phenylethanone	68527-49-1	0.00174	0.00022	
			Sodium sulfate	7757-82-6	0.00137	0.00017	
			Non-crystalline silica (impurity)	7631-86-9	0.00128	0.00016	
			Ethylene Glycol	107-21-1	0.00118	0.00015	
			Alcohols, C14-15, ethoxylated (7EO)	68951-67-7	0.00082	0.00010	
			Ethanol	64-17-5	0.00066	0.00008	
			Propargyl alcohol	107-19-7	0.00055	0.00007	
			2-Propenamid (impurity)	79-06-1	0.00029	0.00004	
			Hexadec-1-ene	629-73-2	0.00018	0.00002	
			Tetrasodium ethylenediaminetetraacetate	64-02-8	0.00015	0.00002	
			Diammonium peroxidisulphate	7727-54-0	0.00008	0.00001	
			1-Octadecene (C18)	112-88-9	0.00009	0.00001	
			Dimethyl siloxanes and silicones	63148-62-9	0.00008	0.00001	
			Decamethyl cyclopentasiloxane	541-02-6	0.00001		
			Siloxanes and silicones, dimethyl, reaction products with silica	67762-90-7	0.00001		
			Octamethylcyclotetrasiloxane	556-67-2	0.00001		
			Formaldehyde	50-00-0	0.00001		
			Dodecamethylcyclohexasiloxane	540-97-6			
			Copper(II) sulfate	7758-98-7			
FR Pro 150	ECM	Friction Reduction					
			Water	7732-18-5	50.00000	0.00240	
			Polyacrylamide-co-acrylic acid	9003-06-9	32.00000	0.00154	
			Sodium Chloride	7647-14-5	15.00000	0.00072	
			Alcohol Ethoxylate Surfactants	Trade	5.00000	0.00024	
			Hydrotreated Petroleum Distillate	64742-47-8	25.00000		

* Total Water Volume sources may include fresh water, produced water, and/or recycled water
** Information is based on the maximum potential for concentration and thus the total may be over 100%

Note: For Field Development Products (products that begin with FDP), MSDS level only information has been provided.
Ingredient information for chemicals subject to 29 CFR 1910.1200(i) and Appendix D are obtained from suppliers Material Safety Data Sheets (MSDS)

Table S6. Hydraulic fracturing fluid composition details for U-6 well in the Utica-Point Pleasant Formation

Job Start Date	4/30/2015
Job End Date	5/30/2015
State	Ohio
County	Monroe
True Vertical Depth	9,619
Total Base Water Volume (gal)	7,519,974
Total Base Non Water Volume	0

Hydraulic Fracturing Fluid Composition:

Trade Name	Supplier	Purpose	Ingredients	Chemical Abstract Service Number (CAS #)	Maximum Ingredient Concentration in Additive (% by mass)**	Maximum Ingredient Concentration in HF Fluid (% by mass)**	Comments
Fresh water	Stingray	Carrier					
			Water	7732-18-5	100.00000	87.27337	
40/70 White	Steubenville/Cadiz	Proppant					
			Sand	14808-60-7	100.00000	11.93720	
Muriatic Acid	Axiall, LLC	Acid					
			Water	7732-18-5	60.00000	0.21773	
			Hydrogen chloride	7647-01-0	40.00000	0.14516	
100 mesh	Minerva/Cadiz	Proppant					
			Sand	14808-60-7	100.00000	0.23238	
FRA 409	Weatherford	Friction Reducer					
			Ethanaminium, N,N,N-trimethyl-2-[(1-oxo-2-propenyl)oxy]-, chloride, polymer with 2-propenamide	69418-26-4	70.00000	0.07463	
			Proprietary	Proprietary	30.00000	0.03198	
			Petroleum Distillate	64742-47-8	10.00000	0.01066	
			Alcohols, C12-14-secondary, ethoxylated	84133-50-6	5.00000	0.00533	
			Adipic acid	124-04-9	3.00000	0.00320	
B-84	X-Chem, LLC	Biocide					
			Water	7732-18-5	55.50000	0.01738	

			Glutaraldehyde	111-30-8	27.00000	0.00846	
			Didecyl dimethyl ammonium chloride	7173-51-5	8.00000	0.00251	
			n-Alkyl dimethyl benzyl ammonium chloride	68424-85-1	5.50000	0.00172	
			Ethanol	64-17-5	4.00000	0.00125	
VBL-29	X-Chem, LLC	Breaker					
			Water	7732-18-5	90.00000	0.02785	
			Hydrogen Peroxide	7722-84-1	10.00000	0.00309	
Plexgel 907L-EB	Chemplex SOLVAY	Viscosifier					
			Guar Gum	9000-30-0	50.00000	0.00638	
			Distillate(petroleum), hydrotreated light	64742-47-8	50.00000	0.00638	
			Organophylic Clay	Proprietary	2.00000	0.00026	
			Alcohol ethoxylate	34398-01-1	0.99000	0.00013	
			Cyrstalline Silica	14808-60-7	0.06000	0.00001	
SC-30	X-Chem, LLC	Scale Inhibitor					
			Water	7732-18-5	70.00000	0.00729	
			Sodium Polyacrylate	Proprietary	30.00000	0.00312	
TCA 6038F	X-Chem, LLC	Corrosion Inhibitor					
			Water	7732-18-5	80.00000	0.00169	
			Methanol	67-56-1	20.00000	0.00042	
Ingredients shown above are subject to 29 CFR 1910.1200(i) and appear on Material Safety Data Sheets (MSDS). Ingredients shown below are Non-MSDS.							

* Total Water Volume sources may include fresh water, produced water, and/or recycled water
** Information is based on the maximum potential for concentration and thus the total may be over 100%

Note: For Field Development Products (products that begin with FDP), MSDS level only information has been provided.
Ingredient information for chemicals subject to 29 CFR 1910.1200(i) and Appendix D are obtained from suppliers Material Safety Data Sheets (MSDS)

Table S7. Hydraulic fracturing fluid composition details for U-7 well in the Utica-Point Pleasant Formation

Job Start Date:	4/30/2015
Job End Date:	5/30/2015
State:	Ohio
County:	Monroe
True Vertical Depth:	9,643
Total Base Water Volume (gal):	7,485,366
Total Base Non Water Volume:	0

Hydraulic Fracturing Fluid Composition:

Trade Name	Supplier	Purpose	Ingredients	Chemical Abstract Service Number (CAS #)	Maximum Ingredient Concentration in Additive (% by mass)**	Maximum Ingredient Concentration in HF Fluid (% by mass)**	Comments
Fresh water	Stingray	Carrier					
			Water	7732-18-5	100.00000	87.34324	
40/70 White	Steubenville/Cadiz	Proppant					
			Sand	14808-60-7	100.00000	11.90548	
Muriatic Acid	Axiall, LLC	Acid					
			Water	7732-18-5	60.00000	0.19527	
			Hydrogen chloride	7647-01-0	40.00000	0.13018	
100 mesh	Minerva/Cadiz	Proppant					
			Sand	14808-60-7	100.00000	0.23254	
FRA 409	Weatherford	Friction Reducer					
			Ethanaminium, N,N,N-trimethyl-2-[(1-oxo-2-propenyl)oxy]-, chloride, polymer with 2-propenamide	69418-26-4	70.00000	0.07695	
			Proprietary	Proprietary	30.00000	0.03298	
			Petroleum Distillate	64742-47-8	10.00000	0.01099	
			Alcohols, C12-14-secondary, ethoxylated	84133-50-6	5.00000	0.00550	
			Adipic acid	124-04-9	3.00000	0.00330	
B-84	X-Chem, LLC	Biocide					
			Water	7732-18-5	55.50000	0.01766	

			Glutaraldehyde	111-30-8	27.00000	0.00859	
			Didecyl dimethyl ammonium chloride	7173-51-5	8.00000	0.00255	
			n-Alkyl dimethyl benzyl ammonium chloride	68424-85-1	5.50000	0.00175	
			Ethanol	64-17-5	4.00000	0.00127	
VBL-29	X-Chem, LLC	Breaker					
			Water	7732-18-5	90.00000	0.02733	
			Hydrogen Peroxide	7722-84-1	10.00000	0.00304	
SC-30	X-Chem, LLC	Scale Inhibitor					
			Water	7732-18-5	70.00000	0.00771	
			Sodium Polyacrylate	Proprietary	30.00000	0.00330	
Plexgel 907L-EB	Chemplex SOLVAY	Viscosifier					
			Guar Gum	9000-30-0	50.00000	0.00435	
			Distillate(petroleum), hydrotreated light	64742-47-8	50.00000	0.00435	
			Organophylic Clay	Proprietary	2.00000	0.00017	
			Alcohol ethoxylate	34398-01-1	0.99000	0.00009	
			Cyrstalline Silica	14808-60-7	0.06000	0.00001	
TCA 6038F	X-Chem, LLC	Corossion Inhibitor					
			Water	7732-18-5	80.00000	0.00117	
			Methanol	67-56-1	20.00000	0.00029	

Ingredients shown above are subject to 29 CFR 1910.1200(i) and appear on Material Safety Data Sheets (MSDS). Ingredients shown below are Non-MSDS.

* Total Water Volume sources may include fresh water, produced water, and/or recycled water

** Information is based on the maximum potential for concentration and thus the total may be over 100%

Note: For Field Development Products (products that begin with FDP), MSDS level only information has been provided.

Ingredient information for chemicals subject to 29 CFR 1910.1200(i) and Appendix D are obtained from suppliers Material Safety Data Sheets (MSDS)

Table S8. Organic Chemistry Data

Type of sample	Well #	Approximate Time After Flowback Began (Days)	Sampling Date	Benzene (µg/L)	Toluene (µg/L)	Ethylbenzene (µg/L)	Xylene t (µg/L)	m,p-xylene (µg/L)	o-Xylene (µg/L)	MBAS (mg/L)	O&G (mg/L)
MARCELLUS SHALE											
M-4 FPW	M-4	1	12/10/2015	3	7.2	0.34	5	4	1.1	0.08	4
M-4 FPW	M-4	13	12/22/2015	2.2	2.7	0.11	1.2	0.71	0.53	0.48	36
M-4 FPW	M-4	42	1/20/2016	10	13	1.1	3.2	2.1	2.3	0.38	32
M-4 FPW	M-4	56	2/3/2016	12	10	11	31	20	10.5	0.55	88
M-4 FPW	M-4	133	4/20/2016	2.8	3.4	0.11	1	0.62	0.41	0.91	2
M-4 FPW	M-4	206	7/2/2016	1.4	1.6	0	0.31	0.2	0.11	0.38	250
M-4 FPW	M-4	287	9/21/2016	9.3	13	0.56	3	1.6	1.3	0.53	8
M-4 FPW	M-4	401	1/13/2017	2.1	2.3	0.11	0.31	0.2	0.11	0.84	2
M-4 FPW	M-4	485	4/7/2017	1.2	0.98	0.11	0.31	0.2	0.11	1.5	1
M-4 FPW	M-4	675	9/20/2017	0.67	0.37	0.2	0.65	0.49	0.18	1.2	2
M-4 FPW	M-4	771	12/20/2017	0.15	0.37	0.2	0.65	0.49	0.18	0.11	1
M-5 FPW	M-5	1	12/10/2015	4.3	18	1.4	16	12	4	0.56	41
M-5 FPW	M-5	8	12/17/2015	14	22	1.1	13	8.1	4.7	0.34	140
M-5 FPW	M-5	13	12/22/2015	12	10	11	31	20	10.5	0.22	41
M-5 FPW	M-5	42	1/20/2016	27	53	4	23	14	9.2	0.26	28
M-5 FPW	M-5	56	2/3/2016	12	10	11	31	20	10.5	0.27	500
M-5 FPW	M-5	133	4/20/2016	5.6	12	1.1	10	7.2	3.2	0.88	4
M-5 FPW	M-5	203	6/29/2016	8.8	15	0.83	3.6	2.1	1.6	0.89	12
M-5 FPW	M-5	287	9/21/2016	1.4	3.2	0.5	0.85	0.46	0.39	0.67	190
M-5 FPW	M-5	402	2/14/2017	2.2	7	0.48	2.1	1.4	0.72	3	16
M-5 FPW	M-5	771	12/20/2017	1.7	1.5	0.2	0.65	0.49	0.18	0.02	7

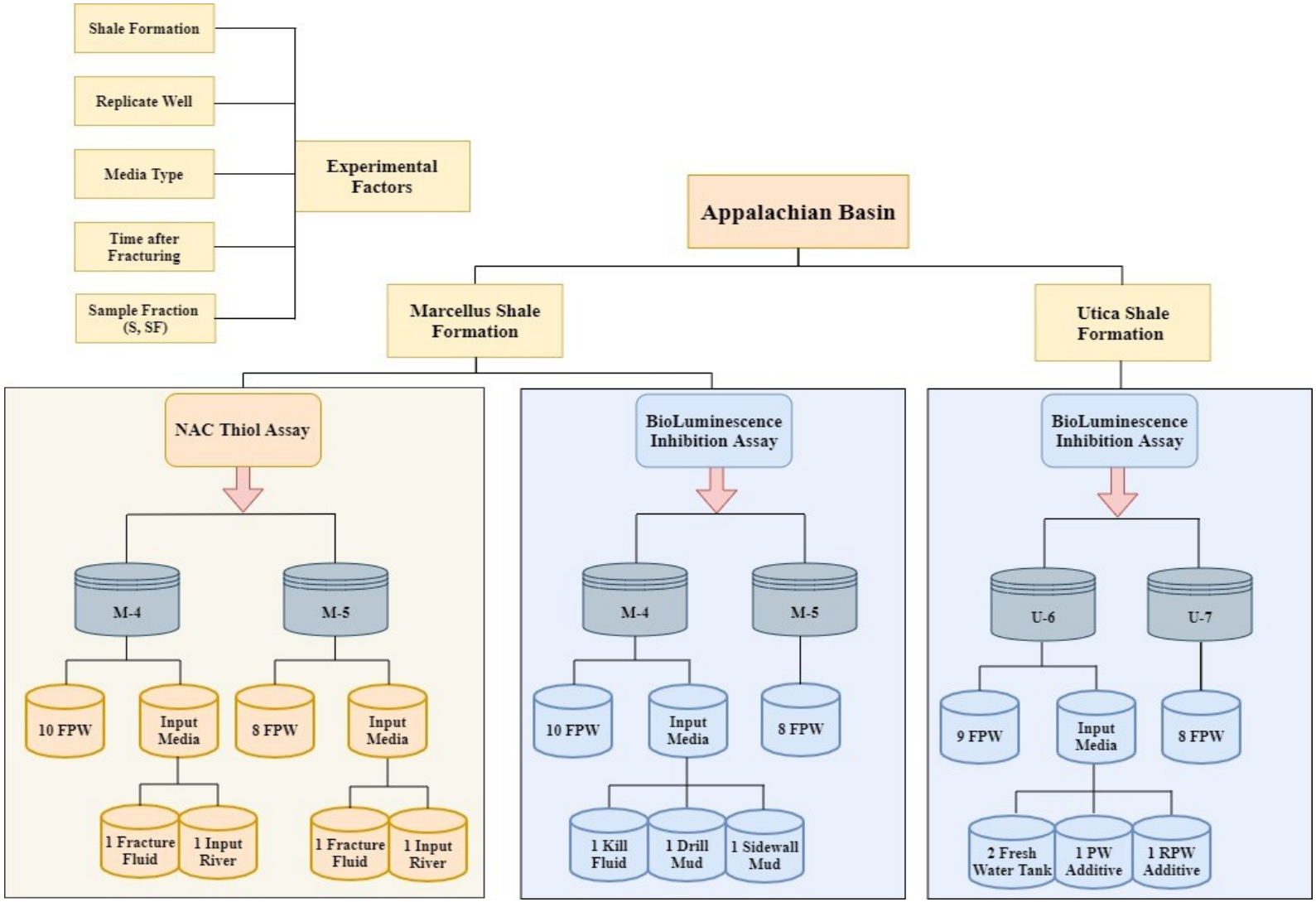
Table S9. Iodinated Organic Ions Data

Type of sample	Well #	Approximate Time After Flowback Began (Days)	Sampling Date	Number of Iodinated Ions	cumulative iodinated intensity
MARCELLUS SHALE					
M-4 FPW	M-4	1	12/10/2015	24	365771652
M-4 FPW	M-4	2	12/11/2015	4	53009405
M-4 FPW	M-4	3	12/12/2015	23	247156272
M-4 FPW	M-4	4	12/13/2015	25	335868550
M-4 FPW	M-4	7	12/16/2015	9	84575542
M-4 FPW	M-4	32	1/10/2016	55	1589292678
M-4 FPW	M-4	52	1/30/2016	54	1232849252
M-4 FPW	M-4	66	2/13/2016	42	701786850
M-4 FPW	M-4	80	2/27/2016	47	1131320476
M-4 FPW	M-4	115	4/2/2016	34	449540466
M-4 FPW	M-4	178	6/4/2016	21	249839235
M-4 FPW	M-4	213	7/9/2016	59	1536697731
M-4 FPW	M-4	276	9/10/2016	28	176118846
M-5 FPW	M-5	1	12/10/2015	38	655626375
M-5 FPW	M-5	5	12/14/2015	12	202148089
M-5 FPW	M-5	27	1/5/2016	27	412030329
M-5 FPW	M-5	47	1/25/2016	36	565581406
M-5 FPW	M-5	61	2/8/2016	31	508076506
M-5 FPW	M-5	110	3/28/2016	43	759333756
M-5 FPW	M-5	271	9/5/2016	34	462125872

Data from J. L. Luek, M. Harir, P. Schmitt-Kopplin, P. J. Mouser and M. Gonsior, Temporal dynamics of halogenated organic compounds in Marcellus Shale flowback, *Water research*, 2018, **136**, 200-206.

II. Supporting Figures

Figure S1. Experimental factors considered in this paper.



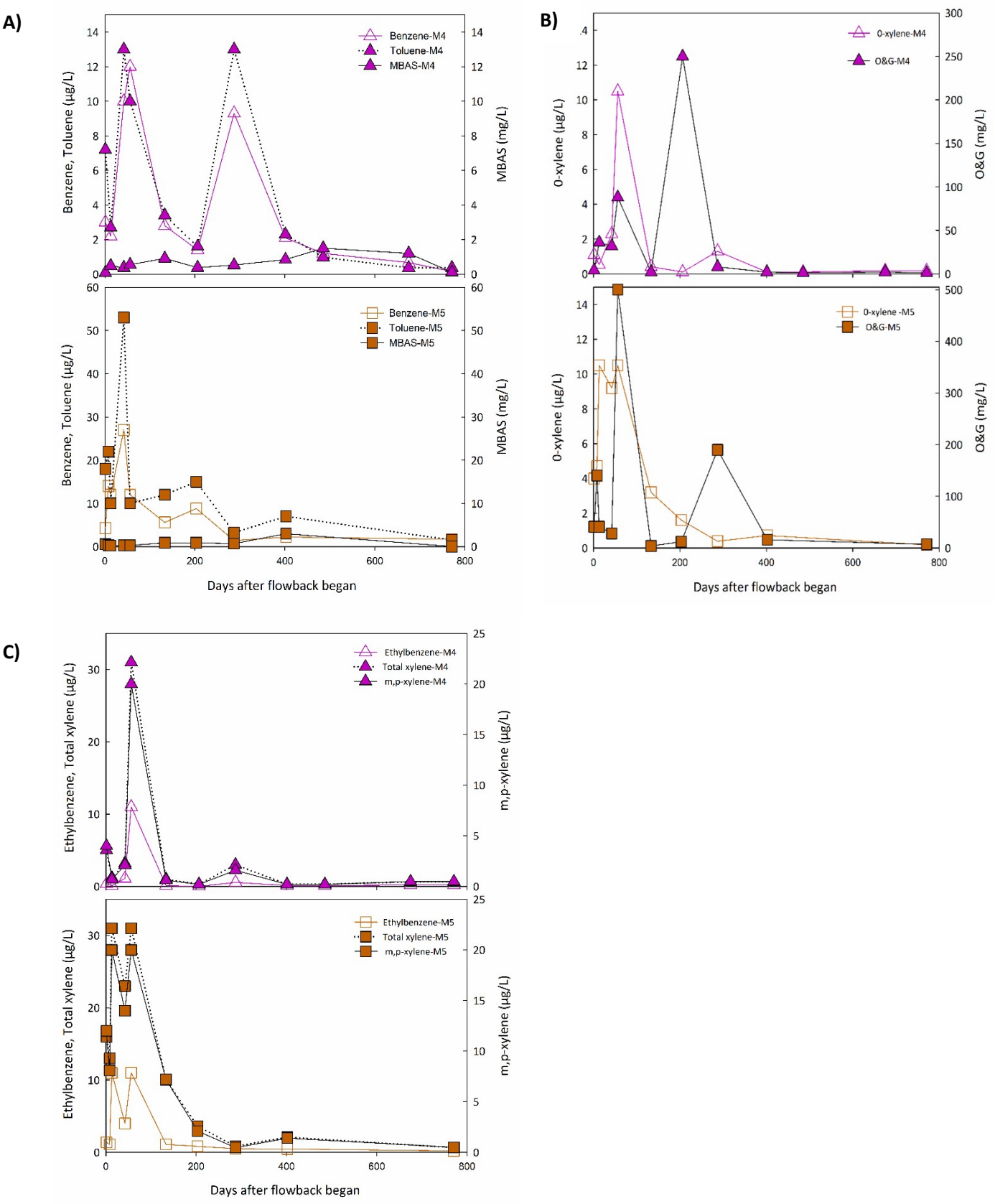


Figure S2.

(A) Benzene, Toluene and MBAS trends, (days 1-771) (B) o-xylene and O&G trends, (days 1-771) and (C) Ethylbenzene, total xylene and m,p-xylene trends in Marcellus shale formation, (days 1-771).